

# Application News

Inductively Coupled Plasma Atomic Emission Spectrometry

No. J92

## Fertilizer Analysis by ICPE-9000

### ■ Introduction

Wastewater disposal sludge, composed of a wide variety of substances, is effectively recycled as fertilizer. However, such recycling requires prior quality confirmation testing of the main constituents as well as the harmful constituents to ensure the safety of the resultant sludge fertilizer. In the case of fertilizer classified as "ordinary fertilizer," the Japan's Fertilizer Control Law specifies an official standard regarding the maximum allowable amounts of harmful substances, and minimum amounts of the necessary principle constituents that should be contained in the fertilizer. The maximum amounts of a harmful components permitted in sludge fertilizer are shown in Table 1.

The method to be used for analysis of fertilizer is specified in the official fertilizer test method (Food and Agricultural Materials Inspection Center), which states that micro-level inorganic compounds are to be

measured quantitatively by atomic absorption following dissolution using the dry ashing method (wet decomposition, in the case of arsenic).

On the other hand, ICP-AES (ICP Atomic Emission Spectrometry) simultaneous multi-element analysis is currently being used in a wide range of applications, and there are expectations for its greater use in fertilizer analysis. Here we introduce an analysis of fertilizer we conducted using the Shimadzu ICPE-9000 Multitype Inductively Coupled Plasma Emission Spectrometer. ICP-AES features a wide dynamic range and high sensitivity, in addition to its applicability to simultaneous multi-element analysis. It is an extremely efficient analytical method, especially when there are many target elements present in a wide concentration range, from the principle constituents to trace level substances.

Table 1 Maximum Allowable Content of Toxic Components (%) in Sludge Fertilizers

Element Name	Arsenic	Cadmium	Mercury	Nickel	Chromium	Lead
Max. Content (%)	0.005	0.0005	0.0002	0.03	0.05	0.01

### ■ Samples

Fermented sludge fertilizer A, baked sludge fertilizer B, ordinary chemical fertilizer C

### ■ Sample Preparation

The powdered 5 g sample is ashed by heating it in a muffle furnace all day and night (about 450 °C). Aqua regia is added, and the sample is heated on a hot plate until just before it dries, and 50 mL hydrochloric acid is then added to dissolve the residue. After cooling to ambient temperature, distilled water is added to adjust the volume to 200 mL. Internal standard elements Y (yttrium) and In (indium) are added to 50 mL of above sample solution, and distilled water is added to adjust the sample solution volume to 100 mL. Separately, prepare a sample consisting of the analyte elements added to the standard solution. This will serve as the spiked recovery test sample.

### ■ Standard Solution for Calibration Curve

A standard solution (1000 ppm) for atomic absorption analysis was prepared, diluting it appropriately with ultrapure water. In addition, as with the sample, the internal standard elements Y (yttrium) and In (indium) were added.

### ■ Analysis

The analytical sample and the spiked recovery test sample were measured using the ICPE-9000. The analytical conditions are shown in Table 2.

### ■ Results

Table 3 shows the semi-quantitation results (%) based on qualitative analysis. Fertilizer contains a large amount of coexisting elements, such as calcium, aluminum, phosphorus, and sulfur, depending on the type. In general, the errors due to interference in the analysis values may occur when there are many coexisting substances in the sample.

Table 4 shows the quantitation results, recovery rate and detection limits using the ICPE-9000. Excellent recovery results were obtained for all elements, indicating that measurement was conducted without adverse influence from the principle components. In addition, the detection limit was less than 1/10 the official specified maximum content, confirming that analysis was conducted with good sensitivity.

Arsenic (As) quantitation value: Due to the possibility of volatilization during pretreatment, this result is taken as a reference value.

Table 2 Analytical Conditions

Instrument	: ICPE-9000
Radio Frequency Output	: 1.2 (kW)
Plasma Gas Flow Rate	: 14 (L/min)
Auxiliary Gas Flow Rate	: 1.2 (L/min)
Carrier Gas Flow Rate	: 0.7 (L/min)
Sample Introduction	: Coaxial nebulizer
Spray Chamber	: Cyclone chamber
Plasma Torch	: Torch for high salt concentration
View Direction	: Axial / Radial

### References

- Official standards for ordinary fertilizer based on the Fertilizer Control Law (Notification No.284 of the Ministry of Agriculture, Forestry and Fisheries, February 22, 1986; Revision Notification No.320 of the Ministry of Agriculture, Forestry and Fisheries, April 9, 2010; Implemented: May 10, 2010)
- Fertilizer Test Methods (2010) (Food and Agricultural Materials Inspection Center)
- JIS K0102: 2008 (Testing Methods for Industrial Wastewater)

Table 3 Semi-Quantitation Results of Fertilizer (Unit: %)

Sample Name	Fermented Sludge Fertilizer A	Baked Sludge Fertilizer B	Ordinary Chemical Fertilizer C
Ca	15	5	7
Al	6	12	0.1
P	2	5	4
S	0.6	0.2	10
Fe	2	1	0.1
K	0.4	1	4
Mg	1	1	0.2
Na	0.1	0.7	1

Table 4 Quantitation Results of Fertilizer (Unit: %)

Element Name	Sample Name		Fermented Sludge Fertilizer A		Baked Sludge Fertilizer B		Ordinary Chemical Fertilizer C	
	Official Max. Content	Detection Limit	Quantitation Value	Recovery Rate (%)	Quantitation Value	Recovery Rate (%)	Quantitation Value	Recovery Rate (%)
As*	0.005	0.00008	0.0002*	98.3	0.0017*	97.8	0.0001*	103.1
Cd	0.0005	0.000001	0.00013	96.0	0.00038	100.3	0.00012	99.6
Cr	0.05	0.000004	0.00455	97.8	0.00466	98.6	0.00189	97.3
Cu	-	0.000003	0.0214	99.7	0.0554	98.9	0.00048	102.5
Ni	0.03	0.000002	0.00283	94.6	0.0110	100.0	0.00046	99.7
Pb	0.01	0.00002	0.0020	96.5	0.0045	101.1	0.0004	98.4
Ti	-	0.0000002	0.0037	101.0	0.0275	-	0.0034	99.7
Zn	-	0.000002	0.0571	95.8	0.208	100.4	0.0039	100.5

Recovery Rate : Recovery rate based on spiked recovery test (%)

Detection limit : Detection limit determined from standard deviation of 5 repeat measurements of calibration curve blank sample (3 $\sigma$ )

\* : Reference value (when wet ashing test method is used)

Semi-quantitation values : Values calculated from ICPEsolution internal database



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